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References

Annis, B. K., and Pedraza, D. F., 1993, "Effect of Friction on Atomic Force Microscopy of Ion Implanted Highly Oriented Pyrolytic Graphite," *Journal of Vacuum Science and Technology, B*, Vol. 11, No. 5, pp. 1759-1765.

Binnig, G., Quate, C. F., and Gerber, Ch., 1986, "Atomic Force Microscope," *Physics Review Letters*, Vol. 56, No. 9, pp. 930-933.

Bogy, D., Yun, X., and Knapp, B. J., 1994, "Enhancement of Head-Disk Interface Durability by Use of Diamond-Like Carbon Overcoats on the Slider's Rail," *IEEE Transactions on Magnetics*, Vol. 30, No. 2, pp. 2148-2153.

Grafstrom, S., Neitzert, M., Hagen, T. Ackermann, J. Neumann, R., Probst, O. and Wortge, M., 1993, "The Role of Topography and Friction for the Image Contrast in Lateral Force Microscopy," *Nanotechnology*, Vol. 4, No. 3, pp. 143-151.

Kaneko, R., Nonaka, N., and Yasuda, K., 1988, "Atomic Force Microscope for Microtribology, *Journal of Vacuum Science and Technology*, Series A, 6(2), pp. 291-292.

Kaneko, R., Miyamoto, T., and Hamada, E., 1991, "Development of a Controlled Frictional Force Microscope and Imaging of Recording Disk Surfaces," *Advanced Information Storage System*, Vol. 1, pp. 267-277.

Kohno, T., Ozawa, N., Miyamoto, K., and Musha, T., 1988, "High Precision Optical Surface Sensor," *Applied Optics*, Vol. 27, No. 1, pp. 103-108.

Marti, O., Colchero, J., and Mlynek, J., 1990, "Combined Scanning Force and Friction Microscopy of Mica," *Nanotechnology*, Vol. 1, No. 2, pp. 145-151.

Marti, O., Colchero, J., and Mlynek, J., 1993, "Friction and Forces on an Atomic Scale," *Nanosources and Manipulation of Atoms under High Fields and Temperature: Application*, Vu Thien Binh, N. Garcia, and K. Dransfeld, eds., Kluwer Academic Publishers, London, UK.

Mate, C. M., McClelland, G. M., Erlandsson R., and Chiang, S., 1987, "Atomical-Scale Friction of a Tungsten Tip on a Graphite Surface," *Physical Review Letters*, Vol. 59, No. 17, pp. 1942-1945.

Mate, C. M., 1992, "Atomic-Force-Microscope Study of Polymer Lubricants on Silicon Surfaces," *Physical Review Letters*, Vol. 68, No. 22, pp. 3323—3326.

Meyer, G., and Amer, N., 1990, "Simultaneous Measurement of Lateral and Normal Forces with an Optical-Beam-Deflection Atomic Force Microscope," *Applied Physics Letters*, Vol. 57, No. 20, pp. 2089-2091.

Miyamoto, T., Kaneko, R., and Andoh, Y., 1991, "Microscopic Adhesion and Friction between a Sharp Diamond Tip and Al_2O_3-TiC", ASME *Advances in Information and Storage Systems*, Vol. 2, pp. 11-21.

Miyamoto, T., Miyake, S., and Kaneko, R., 1993, "Wear Resistance of C^+-implanted Silicon Investigated by Scanning Probe Microscopy," *Wear*, Apr. 13, Vol. 162, pp. 733-738.

Neubauer, G., Cohen, S., McClelland, G., Horne, D., and Mate, C., 1990, "Force Microscopy With Bidirectional Capacitance Sensor," *Review of Scientific Instruments*, Vol. 61, No. 9, pp. 2296-2308.

O'Shea, S. J., Welland, M. E., and Rayment, T., 1992, "Atomic Force Microscope Study of Boundary Layer Lubrication," *Applied Physics Letters*, Vol. 61, No.18, pp. 2240-2242.

Overney, R., and Meyer, E., 1993, "Tribological Investigations Using Friction Force Microscopy," *MRS Bulletin*, May, pp. 26-34.

Ruan, J.-A., and Bhushan, B., 1994, "Atomic-Scale Friction Measurements Using Friction Force Microscopy: Part I-General Principles and New Measurement Techniques," ASME Journal of Tribology, to appear.

Thundat, T., Warmack, R. J., Allison, D. P., Bottomley, L. A., Lourenco, A. J., and Ferrel, T. L., 1992, "Atomic Force Microscopy of Deoxyrbonuoleic Acid Strands Adsorbed on Mica: The Effect of Humidity on Apparent Width and Image Contrast," *Journal of Vacuum Science and Technology*, Series A, Vol. 10, No. 4, pp. 630-635.

Yanagisawa, M., and Motomura, Y., 1987, "An Ultramicro Identation Hardness Tester and Its Application to Thin Films," *Lubrication Engineering*, Vol. 43, No. 1, pp. 52-56.

DISCUSSION

Bharat Bhushan[1]

I would like to congratulate authors for a well written paper. This discusser agrees with authors that most commercial AFM/FFM instruments measure vertical motion and twisting of the tip to measure the normal and friction forces, respectively. This is done to simplify the design and to reduce cost. However, number of lab designs have been published in which lateral and normal forces are independently measured (Erlandsson et al., 1988; Fujisawa et al., 1994).

Authors state that independent measurements of normal and friction force facilitate the calibration and provide more accurate friction data. It is comforting to note that coefficient of friction values reported in this paper are comparable to that obtained using the commercial instruments in which twisting of the tip is used to measure friction forces (Bhushan et al., 1994a, 1994b, 1994c; Bhushan, 1995). Friction response to stepped surfaces and surface slope can also be obtained with a commercial instrument (Overney and Meyer, 1993; Bhushan, 1994, 1995; Bhushan et al., 1994a, 1994b, 1994c). Thus the commercial instruments appear to have no limitations in the friction measurements.

Additional References

Bhushan, B., 1995, "Micro/nanotribological and Its Applications to Magnetic Storage Devices and MEMS," *Tribol. International* (in press).

Bhushan, B., 1995, *Handbook of Micro/Nanotribology*, CRC Press, Boca Raton, FL.

Bhushan, B., Koinkar, V. N., and Ruan, J., 1994a, "Microtribology of Magnetic Media," *Proc. Instn. Mech. Engrs., Part J: J. Eng. Tribol.*, Vol. 208, pp. 17-29.

Bhushan, B., and Ruan, J., 1994b, "Atomic-Scale Friction Measurements Using Friction Force Microscopy: Part II—Application to Magnetic Media," ASME Journal of Tribology, Vol. 116, pp. 389-396.

Bhushan, B., and Koinkar, V. N., 1994c, "Tribological Studies of Silicon for Magnetic Recording Applications," *J. Appl. Phys.*, Vol. 75, pp. 5741-5746.

Erlandsson, R., Hadzioannou, G., Mate, C. M., McClelland, G. M., and Chiang, S., 1988, "Atomic-Scale Friction Between the Muscovite Mica Cleavage Plane and a Tungsten Tip," *J. Chem. Phys.*, Vol. 69, pp. 5190-5193.

Fujisawa, S., Ohta, M., Konishi, T., Sugawara, Y., and Morita, S., 1994, "Difference Between the Forces Measured by an Optical Lever Deflection and by an Optical Interferometer in an Atomic Force Microscope," *Rev. Sci. Instrum.*, Vol. 65, pp. 644-647.

Overney, R., and Meyer, E., 1993, "Tribological Investigations Using Friction Force Microscopy," *MRS Bulletin*, May, pp. 26-34.

Authors' Comments

The discusser's interest in our paper is appreciated.

The authors would like to point out that research in Nanotribology should go far beyond measurements of the nano-friction coefficient. Investigations of nano-indentation hardness, nano-wear, and nano-fatigue wear, together with nano-friction compose the complete world of Nanotribology. The Scanning Probe Microscope (SPM) [including Scanning Tunneling Microscope (STM), Atomic Force Microscope (AFM), Point Contact Microscope (PCM), Friction Force Microscope (FFM), and Lateral Force Microscope (LFM)] provides a practical and reliable tool for these investigations. However, when we studied nano-indentation, nano-wear, and nano-fatigue wear, limitations of many laboratory and commercially designed scanning probe microscopes were exposed: (1) convenient yet reliable experimental calibrations of tip spring constants were not available for sufficiently accurate normal load and lateral friction data, (2) the normal load could not reach a sufficiently large value (typically, the load range from 2 μN to 2 mN is desirable, depending on the tested materials) to damage the tested sample surfaces for nano-indentation, nano-wear and nano-fatigue wear on various films and materials such as Si, ion implanted Si, SiO_2, SiN, SiC, diamond like carbon, Al_2O_3, TiC and Mn-Zn ferrite, and (3) the friction force for such large normal loads with both no-wear and wear was not measurable.

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It is obvious that the new SPM, a modified PCM, presented in this paper adequately meets these challenges: (1) convenient and reliable in-situ experimental calibration techniques are furnished for both spring constants and optical head sensitivities for accurate and comparable normal load and friction force data without using any questionable assumptions or theoretical calculations, (2) a wide range of normal loads is available from a few nN to a few mN due to the advantageous tip assembly design, and (3) friction force can be measured for this large normal load range, so friction force values associated with both wear and no-wear are available. The friction coefficient versus normal load curve presented in this paper for a Si sample, with normal load as high as 7 μN, is a typical example. In many cases, the friction data could be obtained when the normal load was increased to 200 μN (Jiang et al., 1994).

A different type of SPM with a new tip assembly, also modified from the PCM by the authors, with which normal and lateral forces are measured independently, has the same features (Lu et al., 1994b). The authors are not aware of any other similar instruments, including the commercial ones.

With these particular features and functions, one can make a variety of nano-mechanical, nanotribological tests and measurements, such as: (1) nano-indentation hardness tests (Lu and Bogy, 1993a, 1993b; Lu et al., 1994a), (2) nano-friction tests (Bogy and Jiang, 1994; Jiang et al., 1994; Bogy et al., 1995; Miyamoto et al., 1995), (3) nano-wear tests (Miyamoto et al., 1991, 1993; Jiang et al., 1995a), and (4) nano-fatigue wear tests (Bogy and Jiang, 1994).

Using these special techniques, several novel phenomena important to Nanotribology were observed: (1) There are two friction regimes observed when monitoring friction force versus normal load (usually to 200 μN or higher), a low friction regime (friction coefficient is around 0.05) and a high friction regime (friction coefficient is from 0.10 to 0.50, depending on materials). The low friction regime corresponds to a no-wear regime, whereas the high friction regime produces wear. The critical load for transition to wear, for a given tip, represents the intrinsic wear resistance of the tested material (Bogy and Jiang, 1994; Jiang et al., 1994). (2) Two nano-wear mechanisms were clearly identified on thin solid films, one is the removal of material layer by layer, and the other is the film-break-through. Both mechanisms can be used to characterize the wear durability of ultra thin films by either wear depth or critical wear cycle (Bogy et al., 1995; Jiang et al., 1995a). Discoveries of other nano-wear mechanisms are still on-going. (3) Nano-fatigue wear tests show that for 10,000 wear cycles with normal load as large as 20 μN, there is still no wear detectable on some materials, which indicates no-wear sliding is possible. A better understanding of the no-wear sliding conditions is eagerly sought for proper operations of micromachines and hard disk contact magnetic recording. In particular, "negative-wear" (material build-ip on the scanned area with a heavy normal load) on Si samples was found by nano-fatigue wear tests, which discloses that tribochemical phenomena in Nanotribology becomes of practical importance. Friction does not significantly change with the increase of wear cycles during the nano-fatigue wear tests (Bogy and Jiang, 1994).

These observations illustrate the great potential of the PCM (Kaneko et al., 1988) and the two modified PCMs presented in this paper and in a related paper (Lu et al., 1994b), successfully overcoming the limitations of several currently available instruments.

The instruments presented in this and a related paper (Lu et al., 1994b), together with the test techniques and mechanisms provided by the instruments (Bogy and Jiang, 1994), find immediate applications in the fields of micromachines and hard disk magnetic recording for mechanical and tribological characterizations of sub-micron ultra thin films, and fabrication parameter optimization for such films. Several typical examples include: (1) nanotribological characterization of hydrogenated carbon films on Si (Jiang et al., 1995b), (2) nanotribological characterization of carbon films-correlation with Raman spectra (Lu et al., 1995b), (3) nanotribological evaluations of hydrogentated carbon films as thin as 5 nm on magnetic disks (Jiang et al., 1995c), and (4) investigation of the tribological characteristics of SiO_2 films (Miyamoto et al., 1995). Many other important and novel applications are still underway.

The discusser has performed nano-indentation tests and nano-wear tests (without friction data) using the SPM technique (Bhushan et al., 1994a, 1994c). However, contrary to the impression given in his discussion, the tests were not made on commercial instruments. Although a commercial AFM or FFM with a commercial Si_3N_4 tip was used for the friction measurements (with a very light normal load less than 200 nN), a different tip assembly and a different SPM device were used for the nano-indentation and nano-wear tests, apparently due to the inability of the commercial instrument and tip to make nano-indentations and nano-wear marks (which require normal loads much larger than 200 nN). In fact, the different tip used was a diamond tip mounted on a single steel beam supplied by the Kaneko Laboratory, which actually belongs to the PCM tip assembly family including: (1) single-leaf tip assembly (Kaneko et al., 1988; Bogy, 1992; Lu and Bogy, 1993a), (2) parallel-leaf spring tip assembly (Miyamoto et al., 1993; Lu et al., 1994b; Jiang et al., 1995a), and (3) double parallel-leaf spring tip assembly (Lu et al., 1994b; Jiang et al., 1994; Bogy et al., 1995). So the techniques and instrumentation used by the discusser for nano-indentation and nano-wear were actually those of the PCM. Nano-wear tests by the PCM were first proposed and studied by Kaneko and his coworkers (Miyamoto et al., 1991 and 1993), while nano-indentation hardness tests by use of the STM and the PCM were proposed and investigated by Bogy (1992), Lu and Bogy (1993a, 1995a) and Lu et al. (1994a, 1995a), and an algorithm was proposed and developed by Lu and Bogy (1993b) to eliminate the roughness and image shift effect for both nano-indentation tests and nano-wear tests by subtracting the original surface profile from the indented or scratched surface profile.

The discusser has also attempted to solve the thorny calibration problem of the commercial AFM and FFM (Bhushan and Ruan, 1994b). However, the parallel and perpendicular scan techniques proposed by the discusser still rely on the geometry (length, height, and angle) measurement of the tiny tip assembly by both optical microscopes and scanning electron microscopes, and the estimation of the material properties of the tip spring using those of the bulk material, assuming the two are the same. Moreover, the two techniques rely on each other for cross calibration and confirmation, often requiring the use of a special sample. The convenience and reliability of this approach is suspect. In some cases, the values of friction coefficients obtained by parallel scan may be reliable, this is why the friction coefficient data (only in the low friction regimes) obtained by the discusser are comparable with the friction coefficient values reported in this paper. But the reliability and certainty of the calculated values for normal load and friction force are questionable, in particular, when shapes of the SPM tip assemblies are more complicated and normal loads are larger (from 2 μN to 2 mN). In Nanotribology the values of friction coefficient in the low friction regime may not be as important as in Macrotribology to differentiate various tribo-materials because of insensitivity of nano-friction coefficients. The most important parameters are those such as nano-indentation hardness, friction coefficient in the high friction regime, critical load for wear initiation, wear depth under the same test conditions and critical wear cycles for film break-through, which usually require both accurate normal load values and accurate friction force values, not merely friction coefficient data in the low friction regime (Bogy and Jiang, 1994; Bogy et al., 1995; Lu et al., 1995b; Jiang et al., 1994, 1995a, 1995b, 1995c; Miyamoto et al., 1995).

It is concluded that major hardware and software modifications are required for presently available commercial instruments to have comparable performance capabilities with those of the modified PCMs presented in this and the related paper (Lu et al., 1994b).

Additional References

1 Bogy, D. B., 1992, "Surface Modification and Measurement Using a Scanning Tunneling Microscope with a Diamond Tip," ASME JOURNAL OF TRIBOLOGY, Vol. 114, pp. 493–498.

2 Bogy, D. B., and Jiang, Z., 1994, "Nano-Hardness, Nano-Friction and Nano-Wear of Ultra-Thin Overcoats," presented at the MRS Fall Meeting, Boston, MA, Nov. 28–Dec. 2, 1994, to appear in *Thin Films: Stresses and Mechanical Properties (V)*.

3 Bogy, D. B., Lu, C.-J., Jiang, Z., and Miyamoto, T., 1995, "Experimental Determination of Tribological Properties of Ultra-Thin Films," *ZAMP (J. of Appl. Math. and Phys.)*, in press.

4 Lu, C.-J., and Bogy, D. B., 1993a, "Sub-Microindentation Hardness Tests on Thin Film Disks Using a Point Contact Microscope," *Proc. of 1993 JSME International Conference on Advanced Mechanics*, Tokyo, pp. 883–888.

5 Lu, C.-J., and Bogy, D. B., 1993b, "Sub-Microindentation Hardness Tests on Thin Film Magnetic Disks," presented at the ASME Winter Annual Meeting, New Orleans, LA, Nov. 1993, to appear in *Advanced in Information Storage Systems*.

6 Lu, C.-J., Bogy, D. B., and Kaneko, R., 1994a, "Nanoindentation Hardness Tests Using a Point Contact Microscope," ASME JOURNAL OF TRIBOLOGY, Vol. 116, pp. 175–180.

7 Lu, C.-J., Jiang, Z., Bogy, D. B., and Miyamoto, T., 1994b, "Development of a New Tip Assembly for Lateral Force Microscopy and its Application to Thin Film Magnetic Media," presented at the ASME/STLE Tribology Conference, Maui, Hawaii, Oct. 16–19, 1994, to appear in ASME JOURNAL OF TRIBOLOGY.

8 Lu, C. J., and Bogy, D. B., 1995a, "The Effect of Tip Radius on Nano-Indentation Hardness Tests," *Int. J. of Solids and Structures*, in press.

9 Lu, C. J., Bogy, D. B., and Rosenblum, S. S., 1995b, "Nano-tribological Investigations of Carbon Overcoats: Correlation with Raman Spectra," *J. Vac. Sci. Tech.*, in press.

10 Jiang, Z., Lu, C.-J., Bogy, D. B., and Miyamoto, T., 1994, "Dependence on Nano-Friction and Nano-Wear on Loading Force for Sharp Diamond Tips Sliding on Si, Mn-Zn Ferrite and Au," presented at the ASME/STLE Tribology Conference, Maui, Hawaii, Oct. 16–19, 1994, to appear in ASME JOURNAL OF TRIBOLOGY.

11 Jiang, Z., Lu, C.-J., Bogy, D. B., and Miyamoto, T., 1995a, "An Investigation of Experimental Conditions and Characteristics of a Nano-Wear Test," to be presented at the 10th International Conference on Wear of Materials, Cambridge, MA, Apr. 9–13, 1995, to appear in *Wear*.

12 Jiang, Z., Lu, C. J., Bogy, D. B., and Miyamoto, T., 1995b, "Nanotribological Characterization of Hydrogentated Carbon Films by Scanning Probe Microscopy," *Thin Solid Films*, in press.

13 Jiang, Z., Lu, C. J., Bogy, D. B., Bhatia, C. S., and Miyamoto, T., 1995c, "Nanotribological Evaluations of Hydrogenated Carbon Films as Thin as 5 nm on Magnetic Disks," to be presented at the IEEE International Magnetics Conference, San Antonio, Texas, Apr. 18–21, 1995, to appear in *IEEE Transactions on Magnetics*.

14 Kaneko, R., Nonaka, N., and Yasuda, K., 1988, "Atomic Force Microscope for Microtribology," *J. Vac. Sci. and Tech. A.*, Vol. 6(2), pp. 291–292.

15 Miyamoto, T., Kaneko, R., and Andoh, Y., 1991, "Tribological Characteristics of Amorphous Carbon Films Investigated by Point Contact Microscopy," *J. Vac. Sci. Tech.*, Vol. B9, pp. 1338–1339.

16 Miyamoto, T., Miyake, S., and Kaneko, R., 1993, "Wear Resistance of C^+-Implanted Silicon Investigation by Scanning Probe Microscopy," *Wear*, Vol. 162–164, pp. 733–738.

17 Miyamoto, T., Serikawa, T., Hirono, S., Jiang, Z., Bogy, D. B., and Kaneko, R., 1995, "Tribological Characteristics of SiO_2 Films Investigated by Scanning Probe Microscopy," to be presented at the IEEE International Magnetics Conference, San Antonio, Texas, Apr. 18–21, 1995, to appear in *IEEE Transactions on Magnetics*.